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Temperature

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ABSTRACT

Sialons have been evaluated for application as high temperature electrical insulators in contact with molybdenum and tungsten components in hard vacuum applications. Both D.C. and variable frequency A.C. resistivity data indicate the sialons to have electrical resistivity similar to common oxide in the 1000°C or higher range. Metallographic evaluations indicate good bonding of the type 15R AlN polytype to molybdenum and tungsten. The β' or modified silicon nitride phase was unacceptable in terms of vacuum stability. Additives such as MgO which are commonly used to improve densification had a detrimental effect on electrical resistivity. Similar resistivity decreases were produced by additions of molybdenum or tungsten to form cermets. The use of hot pressing at 1800°C with AlN, Al₂O₃ and Si₃N₄ starting powders produced a better product than did a combination of SiO₂ and AlN starting powders. This study indicated that sialons will be suitable insulators in the 1600K range in contact with molybdenum or tungsten if they are produced as a pure ceramic and subsequently bonded to the metal components at temperatures in the 1600K range.

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INTRODUCTION

Exploration of the outer planets of the solar system may include a nuclear electric propulsion system at a power level in the 400 kWe range (Ref. 1). A system which will provide this power at low weight and high reliability utilizes a heat pipe cooled nuclear reactor coupled with thermionic conversion of the heat to electricity.

Each thermionic converter produces approximately one kilowatt of electrical power at 0.6 VDC. A matrix arrangement of converters electrically connected in both series and parallel is required to increase system voltage output and meet reliability requirements. This in turn means that each converter must be electrically insulated from the heat pipe which supplies heat from the reactor to the emitter of the thermionic converter at approximately 1650K. Either molybdenum or tungsten base alloys can be used for the heat input heat pipes and converter emitters, both of which are in contact with the required high temperature insulator.

The general requirements for the insulator on the heat input side of the thermionic converter have been determined by the design constraints of the thermionic power conversion system and are as follows:

- (1) Thermal expansion must be compatible with either molybdenum or tungsten or preferably both since the coefficients of thermal expansion of these two metals are similar.
- (2) Stability in hard vacuum at 1650K for at least seven years, the minimum outer planet mission time.
- (3) Chemical compatibility with molybdenum and tungsten at 1650K.
- (4) Good thermal conductivity to transfer heat from the heat pipe through the insulators to the thermionic converter emitter while retaining electrical insulating properties.
- (5) Maximum electrical resistivity to minimize power losses and electrolysis at the projected maximum operating voltage of 25 VDC.
- (6) Sufficient bonding to both molybdenum and tungsten to minimize the metal/ceramic interface thermal gradients.

In general, carbides are electrical conductors and are therefore not suitable. Single oxides have a high thermal expansion coefficient and are not mechanically compatible with the molybdenum or tungsten alloys. Nitrides are generally unstable in hard vacuum at the temperatures of interest. Sialons (an acronym for silicon, aluminum, oxygen and nitrogen) are a low thermal expansion class of materials which can be viewed either as a modification of nitrides or a modification of oxides of silicon and aluminum. The sialons therefore were selected as a starting point for the development of the needed high temperature insulator since they appeared to meet the electrical and thermal conductivity requirements, had a low thermal expansion coefficient and were

believed to be chemically compatible with molybdenum and tungsten. The addition of oxides to nitrides to form sialons was expected to reduce the nitrogen overpressure requirement thereby making the material stable in vacuum in the 1600K operating range.

PROCESSING METHODS AND STARTING MATERIALS

Sialons can be produced from constituent powders using either sintering or hot pressing in graphite dies to obtain densification. Since a ceramic approaching full density has maximum thermal conductivity, hot pressing which produces a higher density material than sintering was selected as the fabrication process.

The starting materials used for fabrication of the sialons were the following powders:

- Alcoa Type Al6 Aluminum Oxide
- AME Type CP85 Silicon Nitride
- Ceraac Aluminum Nitride 99.9% purity
- American Drug Silicon Oxide 99.9% purity

Chemical analyses for these materials are presented in Table 1.

The powders were ball milled for 16 hours in polyethylene bottles with tungsten carbide balls and tert-butyl alcohol milling fluid. The powder was then dried and sieved. Hot pressing was done in graphite dies using nitrogen as a cover gas.

For the fabrication of cermets, the metal particles were coated with wax then mixed with the ceramic to ceramic coat each metal particle. Additional ceramic was then added to achieve the desired volume percentage of metal in the cermet. The mix was placed in the graphite die and the die and powder was then outgassed at 875K in approximately 10 to 15 mm of nitrogen overpressure. Both the cermet and ceramic samples were hot pressed in the same manner.

The hot pressing cycle used was: 1) heating of the die and powder to the hot pressing temperature, 2) application of the hot pressing pressure, 3) cooling under pressure to 1675K, 4) release of the pressing pressure and 5) cooling to room temperature. Hot pressing temperatures of 1975 to 2150K were evaluated as were hot pressing pressures of 689 N/cm² to 6890 N/cm² (1000 to 10,000 psi) and hot pressing times of 30 to 120 minutes. Hot pressing temperatures were read with an optical pyrometer using 5:1 L/D holraum in the graphite die body.

The β' sialon (equimolecular amounts of Al₂O₃, AlN and Si₃N₄) was found to densify in the 2050 to 2075K range. At temperatures above 2100K this material tended to become porous indicating decomposition at this temperature. Below

1950K inadequate reaction among the powders also resulted in a poor density material. The 15R polytype material (higher AlN contents) displayed optimum densification in the 2075 to 2100K range.

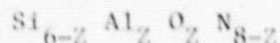
Pressures in the 1378 N/cm² (2000 psi) to 2756 N/cm² (4000 psi) range produced good densification of the sialons while pressures higher than this resulted in fracture of the samples when the pressure was released. Pressure reduction from about 3445 N/cm² (5000 psi) to 1378 N/cm² (2000 psi) at the 2075K hot pressing temperature in an attempt to stress relief anneal the β' sialon also resulted in extensive cracking of this material. This effect was not evaluated with the 15R sialon and the release of pressure at 1675K was used with all materials.

Hot pressing time was the least critical of the variables. The 30 minute pressing cycle produced erratic results while no differences were observed between the 60 and 120 minute cycles. The 60 minute cycle was therefore retained.

The samples tested for electrical resistivity were all hot pressed at 2075K for 60 minutes at a pressure of 1378 N/cm² (2000 psi) in graphite dies using a N₂ cover gas at atmospheric pressure.

A series of phase diagrams have been published on the Al₂O₃, AlN Si₃N₄ and SiO₂ sialon systems (Refs. 2,3,4). The latest of these is by Jack (Ref. 5) (Figure 1) and is generally in agreement with this work. One continuing problem both in determination of an accurate phase diagram and in preparation of quality material is the existence of second phases from both impurities and metastable phases. These inhomogeneities are known to produce adverse effects on the mechanical properties of the ceramic since inhomogeneities are usually not well bonded to the ceramic matrix and as a consequence act as a defect or stress risers. The amount of second phase can be affected by impurities, starting material and fabrication cycle. This is illustrated by fabrication of β' sialon as discussed in the following paragraphs.

β' is described by the formula:



where Z is less than 4.2. Setting Z = 3 yields Si₃N₄·Al₂O₃·AlN. The equivalent material can also be produced from Si₃N₄·3SiO₂·6AlN. Photomicrographs of material produced in these two ways are shown in Figures 2 and 3. The structures are similar and show small amounts of second phase. Microprobe analyses for silicon and aluminum show that the ratio of Al to Si in the sample prepared from SiO₂, Si₃N₄ and AlN varies from 55:45 to 45:55 with some areas being 1:1 which is the predicted ratio for the specified β' . This would indicate the formation of two metastable phases, X-phase which is silicon dioxide rich and the 15R phase which is aluminum nitride rich, as well as β' . The same material fabricated from Si₃N₄, Al₂O₃ and AlN showed much less inhomogeneity.

The implication of this observation is that X-phase (which has not been evaluated in this study) may remain in β' even when fabricated from $\text{AlN} \cdot \text{Al}_2\text{O}_3 \cdot \text{Si}_3\text{N}_4$ since SiO_2 is present as a layer on Si_3N_4 .

The effects of SiO_2 have been shown in the fabrication of the 15R polytype material. This material again can be fabricated either as $\text{SiO}_2 \cdot 4\text{AlN}$ or as $\text{Si}_3\text{N}_4 \cdot 2\text{Al}_2\text{O}_3 \cdot 8\text{AlN}$. Using the same powder processing techniques and hot pressing cycle, the AlN/SiO_2 material was much less dense than that fabricated from $\text{Si}_3\text{N}_4/\text{AlN}/\text{Al}_2\text{O}_3$ as can be seen by comparison of Figures 4 and 5. Microprobe testing of this material indicated both to be relatively homogenous with the predicted aluminum/silicon ratio of 4:1.

ELECTRICAL RESISTIVITY TESTING METHODS

The objective of the fabrication development effort was to produce a high quality sialon material for electrical insulators meeting the requirements outlined in the introduction. The effects of composition and processing on vacuum stability were evaluated as the first step in selecting a material for fabrication and test in prototypical insulator geometry for Nuclear Electric Propulsion (NEP) system application. The second step in the selection process is the measurement of electrical resistivity of the sialons also as a function of composition and processing. The results of this testing are presented in the following section.

Electrical resistivity testing was carried out in hard vacuum (10^{-6} torr or better) using a tungsten mesh heating element and heat shields. A sketch of the setup is shown in Figure 6. DC measurements were taken with a variable voltage megohmmeter and a digital multimeter. AC measurements were taken with an oscillator and an impedance bridge. Samples were 1.9 cm (3/4 in.) square and 0.2 cm (0.080 in.) thick. The electrodes were tungsten or molybdenum foil 0.005 cm (0.002 in.) thick. During initial heating, readings were scattered due to lack of contact between the foil electrodes and the samples. After reaching temperature and holding, the creep produced by the weight resulted in good contact between the electrodes and the samples resulting in reproducible readings. Readings were taken on silicon nitride and Lucalox alumina using this method and agreed well with the published literature values (Figure 7).

Three types of samples have been tested: 1) pure ceramics, 2) cermets using both molybdenum and tungsten metal phases and 3) cermets bonded between molybdenum plates. Each of these types of materials and the electrical resistivity results are discussed in the following sections of this report.

ELECTRICAL RESISTIVITY TEST RESULTS

A. β' SIALON CERAMIC AND CERMETS

The initial materials tested in the program were the β' ceramics. As discussed previously this material contains small amounts of second phases from impurities in the starting powders and as a result of the powder ball milling

process. Free silicon metal was also present apparently due to incomplete nitriding of the silicon to form silicon nitride. Resistivity tests on β' containing equimolecular amounts of Al_2O_3 , AlN and Si_3N_4 displayed erratic behavior at elevated temperatures as indicated in Figure 7. Post test examination of the ceramic indicated weight loss apparently due to decomposition and sublimation of this material at temperatures above 1500K.

Molybdenum cermets made with β' indicated reaction between the molybdenum and ceramic. This reaction can be seen in Figure 8 as the formation of a silicide layer on the surface of the molybdenum particles. There was also evidence of separation between the molybdenum and the ceramic indicating a poor thermal expansion match. Electrical resistivity test results on a tungsten/ β' cermet produced data very similar to pure Si_3N_4 (Figure 7). This was a slightly higher resistivity than was reported by Thorpe and Sharif (Ref. 6). The fact that the cermet test result did not obey Maxwell's mixing rule (Ref. 7) may be due to either the poor bond between the metal and ceramic or due to chemical interaction between the metal and ceramic resulting in reduction in ions which were available for electrical conduction. Since any of the above problem areas would eliminate the material from consideration for application as a high temperature insulator, emphasis therefore was placed on 15R polytype sialon phase.

B. 15R SIALON POLYTYPE

As indicated by the phase diagram of Figure 1, the 15R polytype sialon phase exists over a range of compositions. Initial resistivity testing was done on a composition near the center of the single phase region. This material was hot pressed from Al_2O_3 , AlN and Si_3N_4 as a ceramic disc. Samples 18 mm square by 2 mm thick were machined from the ceramic and tested between molybdenum foil electrodes as previously discussed. Results of electrical resistivity testing of 15R polytype sialon with a composition near the center of the single phase region are plotted in Figure 9. The shape of this electrical resistivity curve is typical of all variations of the 15R polytype ceramic in that a deviation from linearity occurs in the 1400K range, and is discussed in the section on Data Analysis.

Variation of composition within the single phase 15R region had essentially no effect on electrical resistivity. 15R polytype sialon fabricated with the maximum silicon nitride content as defined by the phase diagram (Figure 1) had identical resistivity as the 15R composition in the middle of the single phase region. Results of electrical resistivity testing of the 15R polytype sialon with maximum Si_3N_4 content are plotted in Figure 10. The discontinuity was still present in the resistivity curve in the 1400K range. Resistivity data taken using AC current are also presented in this plot. At high temperatures there is no effect of frequency on electrical resistivity. As temperature decreases the highest frequency AC data are the first to deviate from the DC curve, as has been reported for other ceramics (Ref. 6).

Variation of starting materials, as discussed in previous sections, in turn affected density and probably the amount and distribution of metastable phases within the sialon ceramic although the final chemical compositions were identical. The 15R polytype sialon can be fabricated from SiO_2 and AlN as well as from AlN , Al_2O_3 and Si_3N_4 . The effect of this variation can be observed by comparison of Figures 9, 10 and 11. Figures 9 and 10 summarize data on 15R polytype materials fabricated from Al_2O_3 , AlN and Si_3N_4 . Figure 11 shows electrical resistivity of 15R material fabricated from SiO_2 and AlN . The slope of curve in Figure 11 has changed and the discontinuity in the 1400K range has virtually disappeared. Possible causes of this will be discussed in the following section.

Magnesium oxide is a common addition made to silicon nitride to increase densification rates during sintering or hot pressing. The effect of this additive on electrical resistivity of 15R polytype sialon is shown in Figure 12. The 1400K discontinuity is similar to the same material without the MgO as plotted in Figure 9. However, the resistivity both above and below 1400K has been markedly reduced by the MgO addition. This will also be discussed further in the following section.

To simulate a possible fabrication method for the insulators, molybdenum plates were plasma sprayed with tungsten. The tungsten plates provided a roughened surface to improve bonding of the ceramic. The powder to be formed was placed between these plates and the assembly placed in the graphite dies. The sandwich structure was then hot pressed to consolidate the ceramic to a layer approximately 2 mm thick. Test results on this sandwich structure (Figure 13) indicated the resistivity as a function of temperature to be linear, but at one to two orders of magnitude lower resistivity than sialon fabricated as a ceramic and not in contact with a refractory metal during the hot pressing process. The decreased resistivity may partially be due to improved electrical contact between the sample and the test electrodes. However, the majority of the decrease is believed due to chemical interaction between the refractory metal and the sialon or the starting ceramic powders at hot pressing temperatures prior to formation of sialon. The implication of this observation is that the ceramic should be fabricated to the desired shape at the 2100K hot pressing temperature, then bonded to refractory metal at a temperature near the use temperature (1675K) to minimize the interaction and the resulting resistivity decrease.

A section of a prototypical insulator bonded in this manner is shown in Figure 14.

DATA ANALYSIS

The single characteristic that is common to most of the electrical resistivity data is the change in slope of the resistivity versus temperature curve in the 1400K range.

This change of slope implies a change of activation energy and therefore a change in conducting mechanism for the 15R polytype sialon materials.

The activation energies measured in this work for the 15R polytype sialon (Figures 9, 10 & 12) are 1.8 to 2.4 eV below 1400K and 2.8 to 3.5 eV above 1400K.

Previous investigations of the electrical conductivity of AlN have obtained activation energies of 1.9 eV (Ref. 8) and 1.83 eV (Ref. 9). Both authors agreed that the low activation energy was due to the extrinsic electronic conductivity resulting from carbon contamination. Gorbator et al (Ref. 8) have observed n-type electronic conductivity in carbon contaminated AlN. Francis et al (Ref. 9) have observed a change in conductivity with varying carbon content in AlN.

The band energy gap, E_g , was reported to be between 5.7 to 6.2 eV for AlN (Refs. 10, 11). These values are in reasonable agreement with the absorption values, 4.8 ~ 5.9 eV, measured optically for AlN single crystals (Ref. 12). The intrinsic electronic conduction would thus require a much greater activation energy ($\sim E_g/2 = 2.4$ to 3.1) than that is deduced from the slope of the Arrhenius plots for sialons at low temperatures (1.8 to 2.4 eV) (Figures 9, 10 & 12).

The 15R polytype sialons in this work were hot pressed in graphite dies at high temperature. Since the activation energy obtained below ~ 1350K is roughly that obtained for contaminated AlN, it is quite possible that the DC conductivity of 15R polytype is dominated by extrinsic electronic conduction mechanism produced by carbon contamination.

This view is confirmed by the data in Figure 11, the resistivity of 15R pressed from $\text{SiO}_2 + \text{AlN}$. There is no change of slope in this curve indicating that carbon contamination is minimal in this sample. This can be explained by the probable reaction between the SiO_2 and the graphite die:



At the hot pressing temperature this reaction proceeds to the right with a free energy of -14,000 cal/mole. Thus carbon contamination of the sialon is replaced by formation of a SiC reaction layer. In addition the porosity of this sample could be partially due to the presence of CO- the equilibrium pressure being in the 100 atmosphere range or 2000 psi, the hot pressing pressure.

When the sialon was hot pressed between molybdenum plates the molybdenum minimized carbon pickup by the sialon. This also eliminated the change in slope of the resistivity curve (Figure 13).

In typical covalent semiconductors, it is common to have extrinsic electronic conduction to predominate at lower temperatures whereas the intrinsic electronic conduction becomes dominant at high temperature range when the valence electrons of majority lattice atoms (rather than impurity atoms) gain sufficient thermal energy to be raised into the conduction band. In the case of AlN, the resulting activation energy will be $(E_g)_{\text{intrinsic}}/2 \approx 1/2$ (5.7 to 6.2) eV ≈ 2.85 to 3.1 eV. This is within the 2.8 to 3.5 eV range obtained in this work for high AlN content sialon, i.e., the 15R polytype material.

It would appear that in the high temperature region, the electrical conduction is dominated by the intrinsic electronic mechanism, determined by the band gap, as in the case of classic semiconductors. Two phenomena have been observed, however, which make the electronic conduction model questionable. First, the DC resistivity values were found to drift with time at a constant applied voltage. Secondly, the applied voltage across the measuring electrodes, when increased, increased the apparent DC resistivity of the sialon. Both of these characteristics indicate the possible occurrence of electrolytic polarization process under the applied voltage, rather than a dielectric polarization. Electrolytic polarization involves the ionic dissociation within the material and tends to build up a counter EMF as a result of a translational motion (diffusion) of these dissociated ions over large distances (under the applied voltage). The resistivity of 15R polytype sialon was reduced by the addition of MgO (Figure 12). However, the slope of the resistivity plot remained unchanged from that of the identical material without the MgO addition (Figure 10). This implies that the major conducting mechanism both with and without the MgO addition, is the same. Minority ions such as Mg appear to be the most likely conductors.

The activation energy values obtained for 15R polytype sialon (2.8 eV to 3.5 eV) are greater than those obtained in low temperature range (1.8 eV to 2.4 eV), but lower than that obtained for β' sialon (as high as 4.4 eV) in the same temperature range.

It is known that the β' sialon has the ability to incorporate impurity atoms into the crystal lattice (Ref. 5). This appears to indicate that the β' phase can restrict impurity movement or diffusion producing the higher activation energy observed for this material. Impurity atoms are not as readily incorporated into the AlN type lattice of the 15R polytype material; hence the activation energy measured for the 15R as well as the resistivity is lower than for β' sialon.

Definition of the exact conduction mechanism which operates in sialon was not the objective of this work. The objective was rather to determine if sialon is a candidate for high temperature electrical insulator applications. At present the answer to this question appears to depend upon preparation of higher purity sialon materials coupled with long term testing in vacuum under applied DC potential to evaluate deterioration of sialon under these conditions. Such testing is presently being initiated.

SUMMARY

Electrical resistivity testing of sialons in hard vacuum at elevated temperatures has indicated the 15R polytype to be a candidate for high temperature electrical insulator application in contact with molybdenum or tungsten. β' sialon does not have the required vacuum stability nor an adequate thermal expansion match to molybdenum. The 15R polytype material has adequate electrical resistivity, good vacuum stability and good thermal expansion match to tungsten and molybdenum.

Optimum properties are achieved in the 15R polytype sialon by minimizing the presence of elements other than silicon, aluminum, nitrogen and oxygen. Fabrication at 2075K in contact with refractory metals has a deleterious effect on resistivity; hence, fabrication of a pure ceramic with subsequent bonding to molybdenum or tungsten at a lower temperature is required.

The work presented here is the initial step in developing a high temperature insulator. The major concern remaining is the effects of long term operation under applied electrical load or potential. The degradation mechanism under these conditions is believed to be solid state electrolysis caused by impurity atoms. Additional testing has been initiated to evaluate these effects on the sialon material. Results of this follow-on effort will define voltage and temperature effects on the material as well as define any electrolytic movement of elements within the ceramic. Fabrication methods for bonding the ceramic to molybdenum or tungsten in prototypical geometries and long term evaluation of prototypical units have also been initiated.

ACKNOWLEDGEMENT

The authors would like to express their appreciation to Dr. F.A. Kroger of UCLA who has provided consultation during performance of this work. Also the work of W.C. Estabrook and D.V. Bacigalup in the fabrication and testing of the material is gratefully acknowledged.

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Table 1. Chemical Analyses of Starting Powders
in Weight Percent

Element	Alcoa A16 Al_2O_3	AME CP85Si ₃ N ₄	Cerac AlN	Am Drug SiO ₂
Al	Major	1.0	Major	0.5
Cr	N.D.	0.01	0.01	N.D.
Cu	N.D.	0.02	0.001	0.001
Fe	0.001	0.5	0.2	0.008
Ca	N.D.	0.2	Not determined	N.D.
Ni	N.D.	0.01	0.01	N.D.
Mg	0.02	0.01	0.001	0.01
Mn	N.D.	0.03	0.02	N.D.
Si	N.D.	Major	0.1	Major
O	Major	1.77	2.34	Major
N.D. Not Detected				

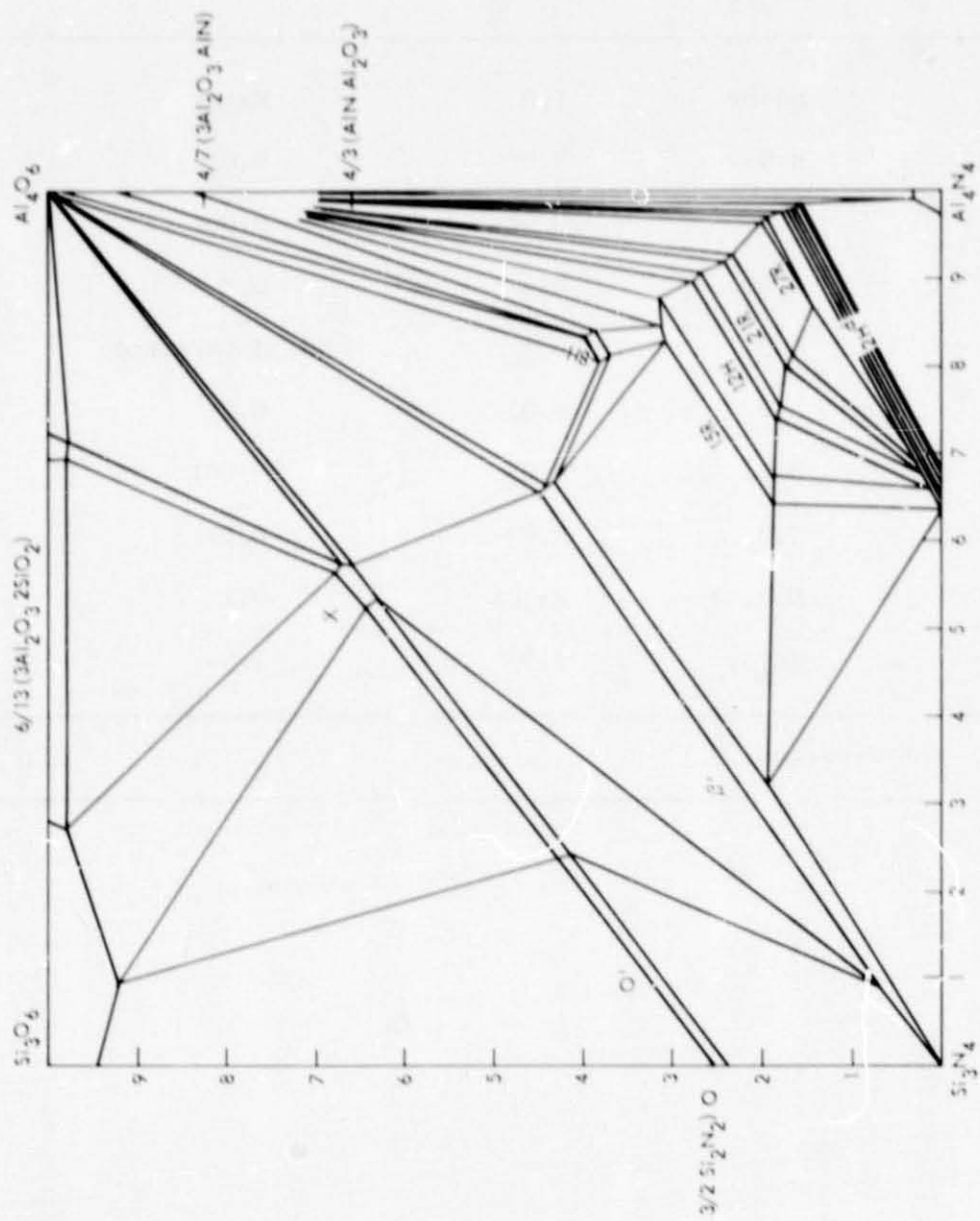
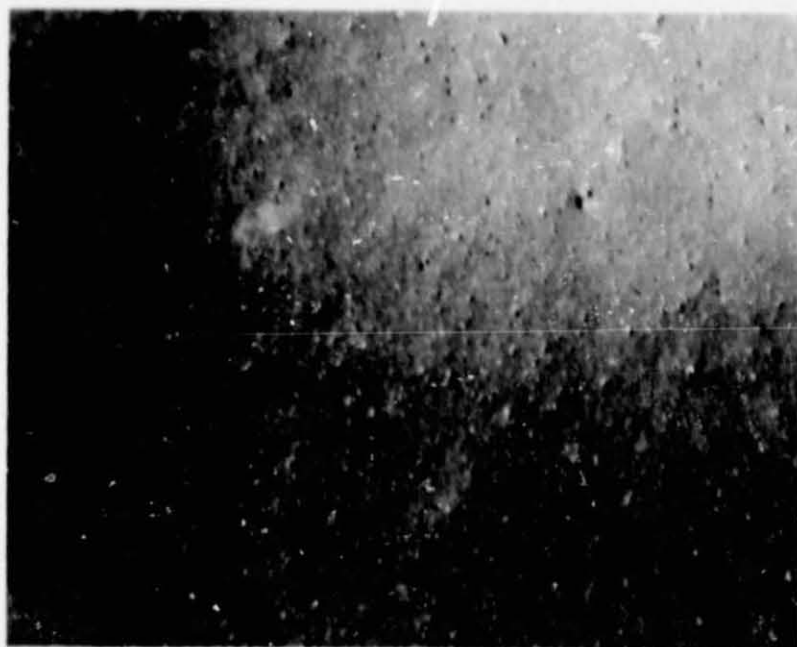


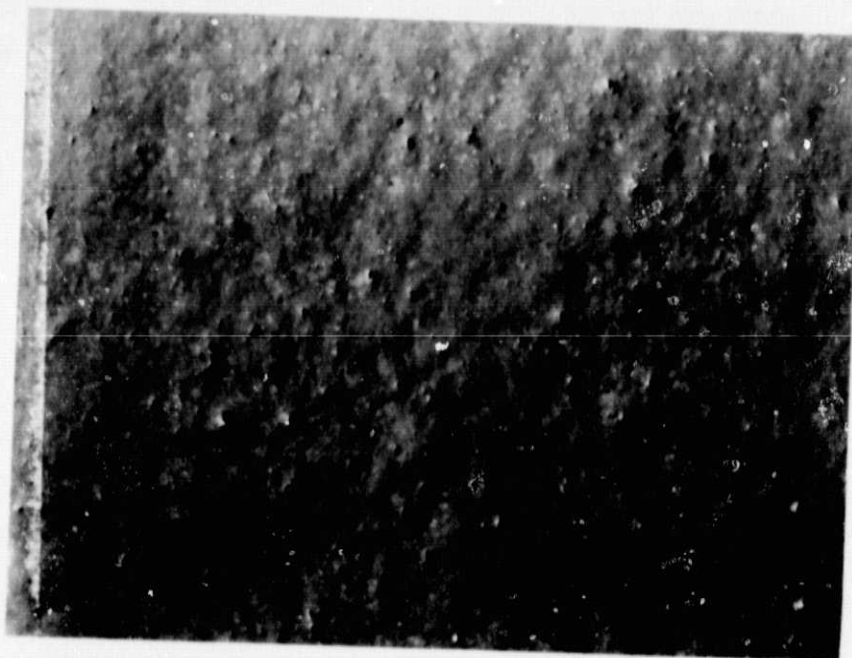
Figure 1. Phase Diagram of the Si_3N_4 - AlN - Al_2O_3 - SiO_2 System



100X

Figure 2. β' Sialon Fabricated by Hot Pressing
of AlN , Al_2O_3 and Si_3N_4

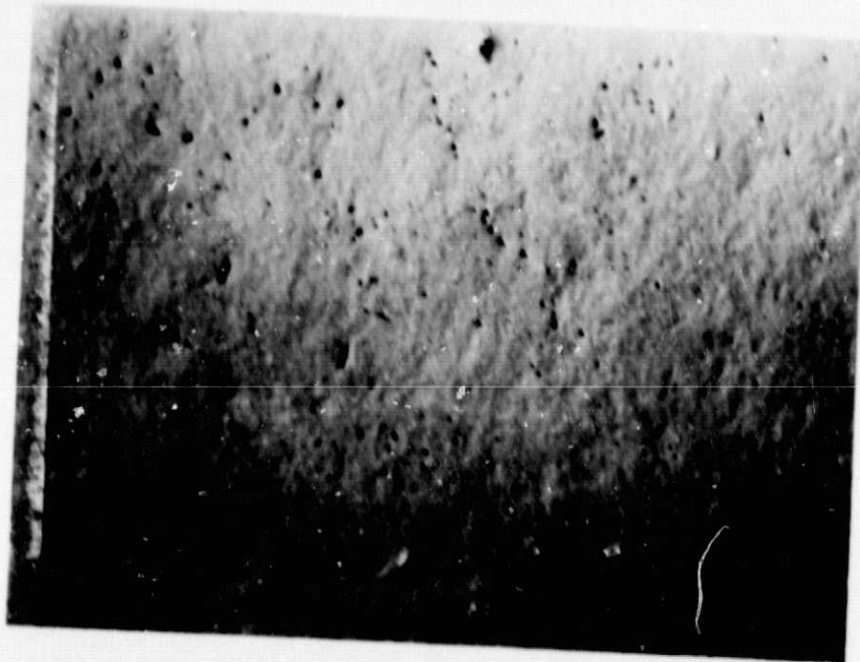
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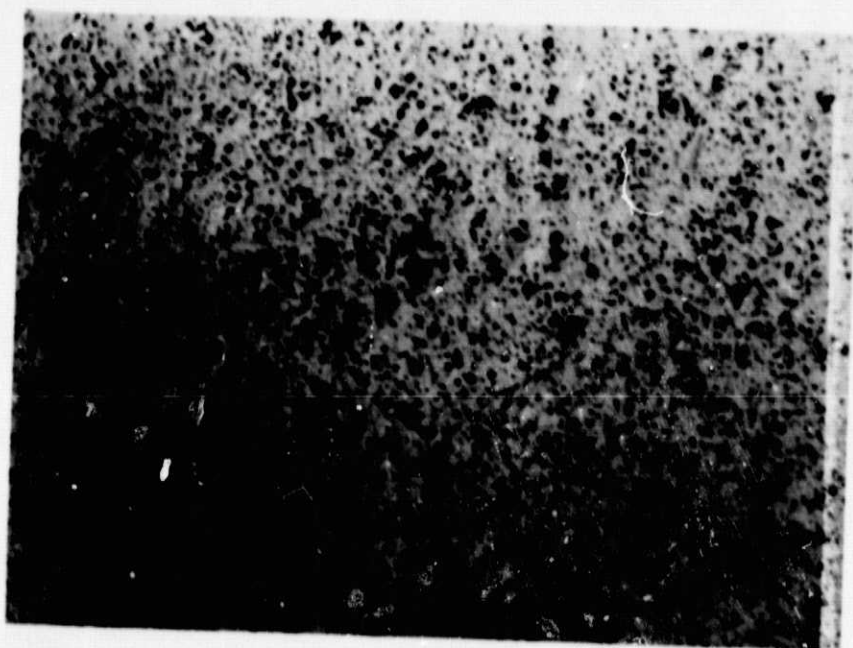
Figure 3. β' Sialon Fabricated by Hot Pressing
of AlN, SiO₂ and Si₃N₄

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Figure 4. 15R Polytype Sialon Fabricated by Hot
Pressing of AlN , Al_2O_3 and Si_3N_4



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Figure 5. 15R Polytype Sialon Fabricated by Hot Pressing of AlN and SiO₂

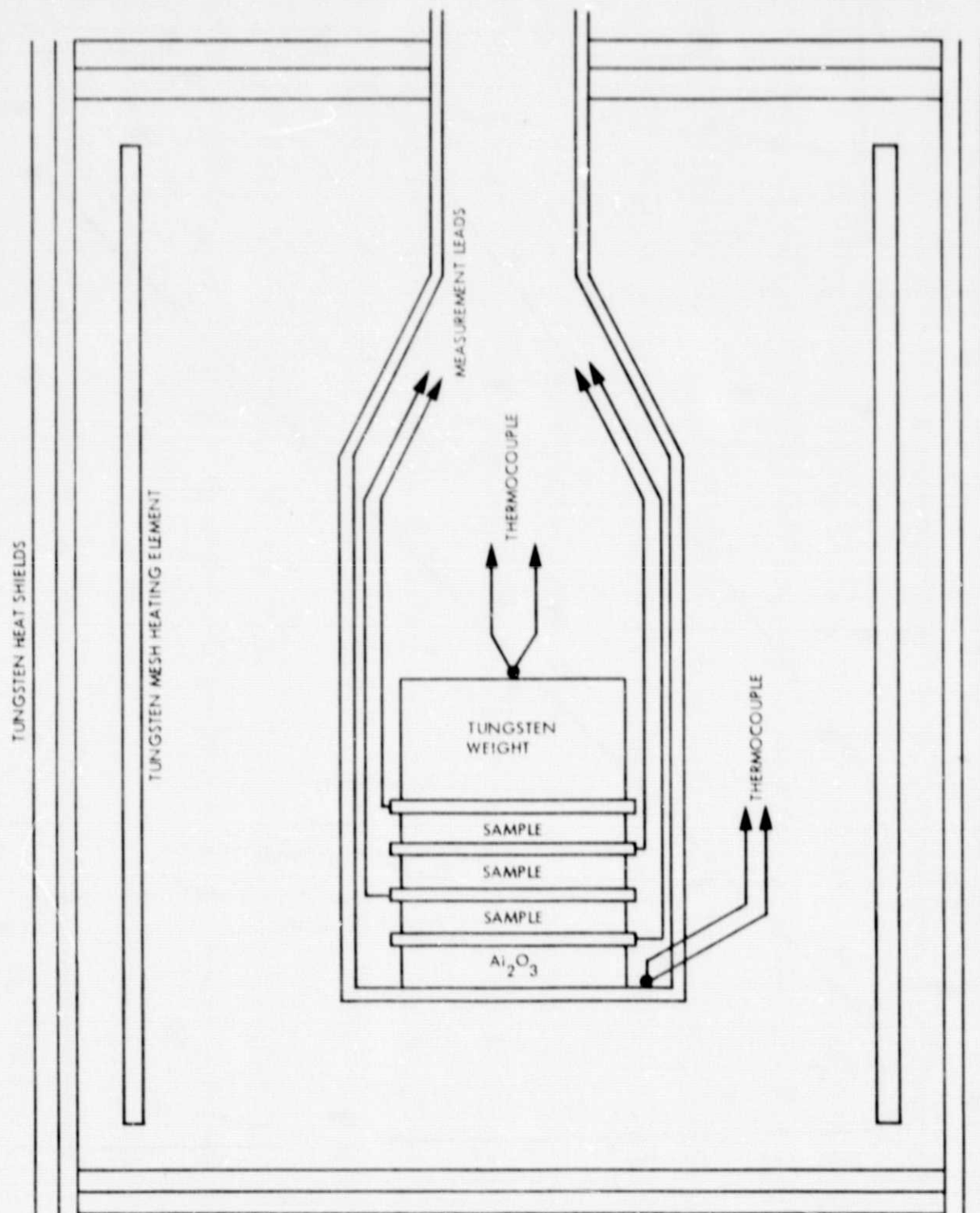


Figure 6. Schematic of Electrical Resistivity Measurement Apparatus

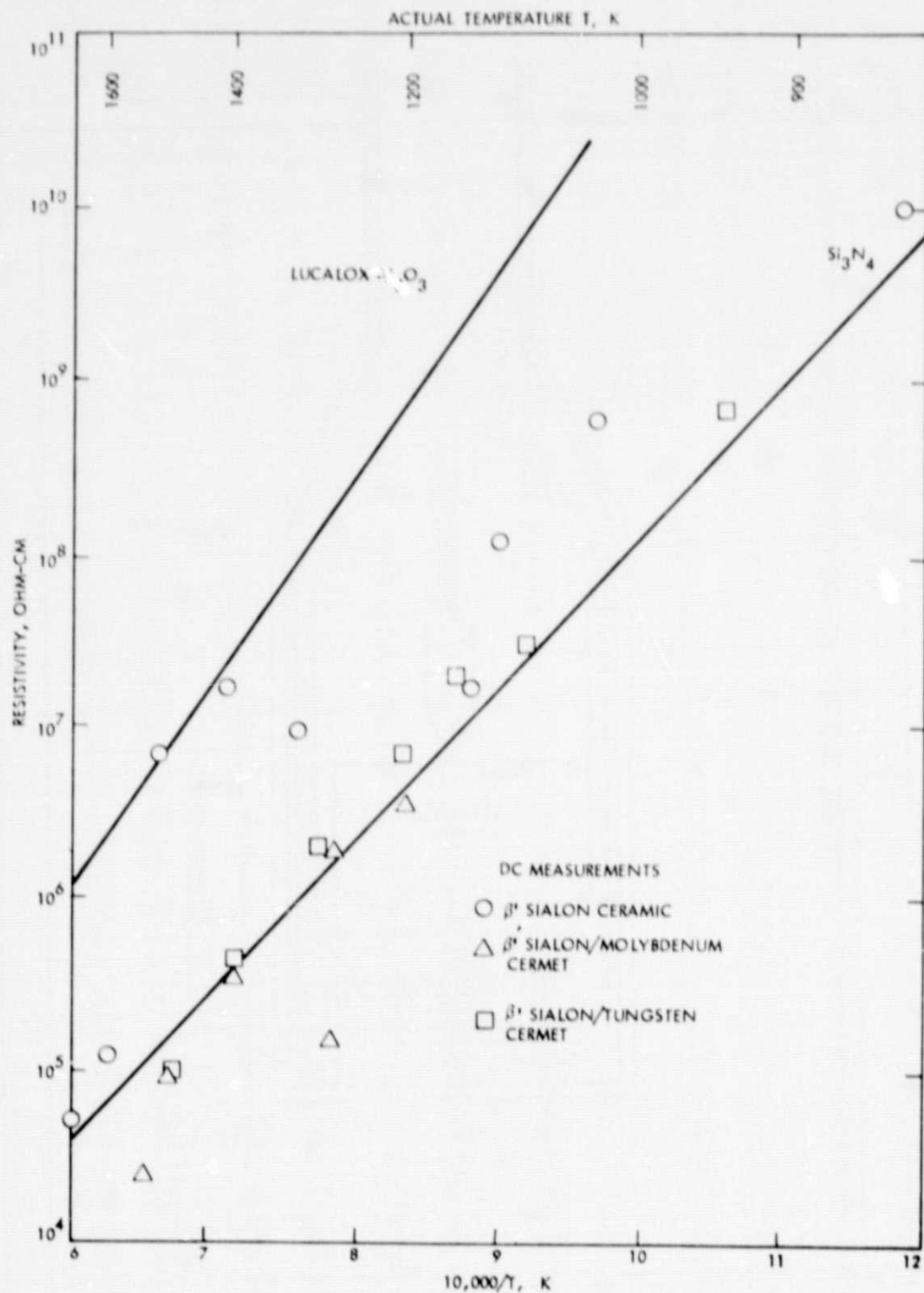
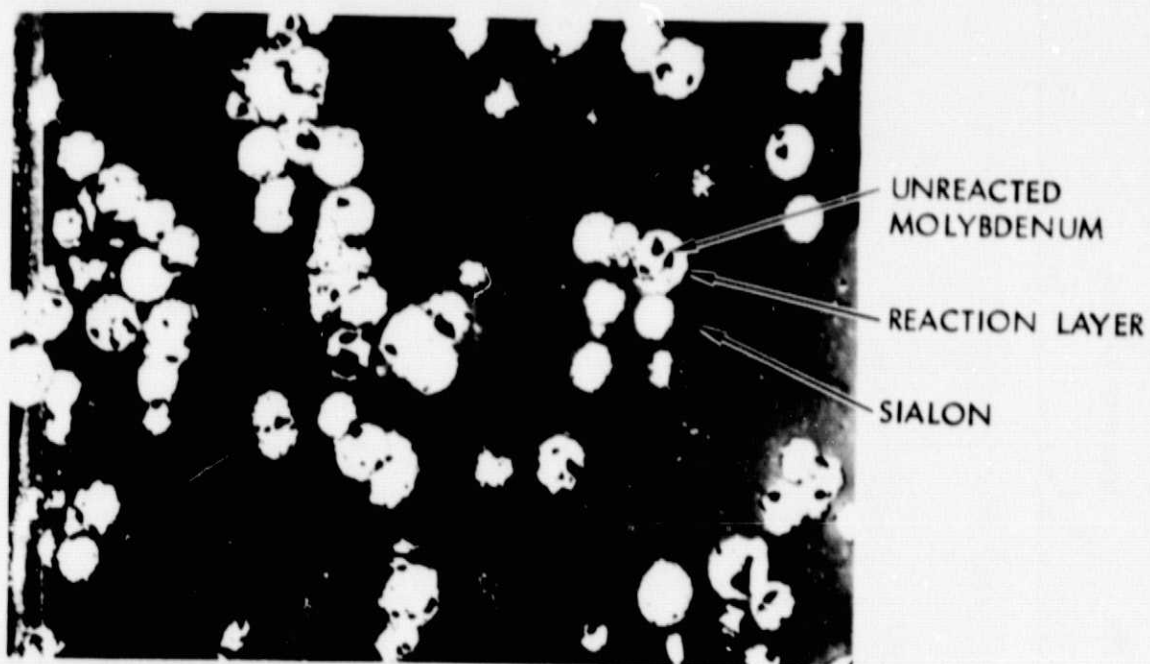


Figure 7. Electrical Resistivity of Lucalox Alumina, Norton 132 Silicon Nitride and β' Sialon Ceramic and Cermets

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Figure 8. β' Sialon/Molybdenum Cermet Microstructure

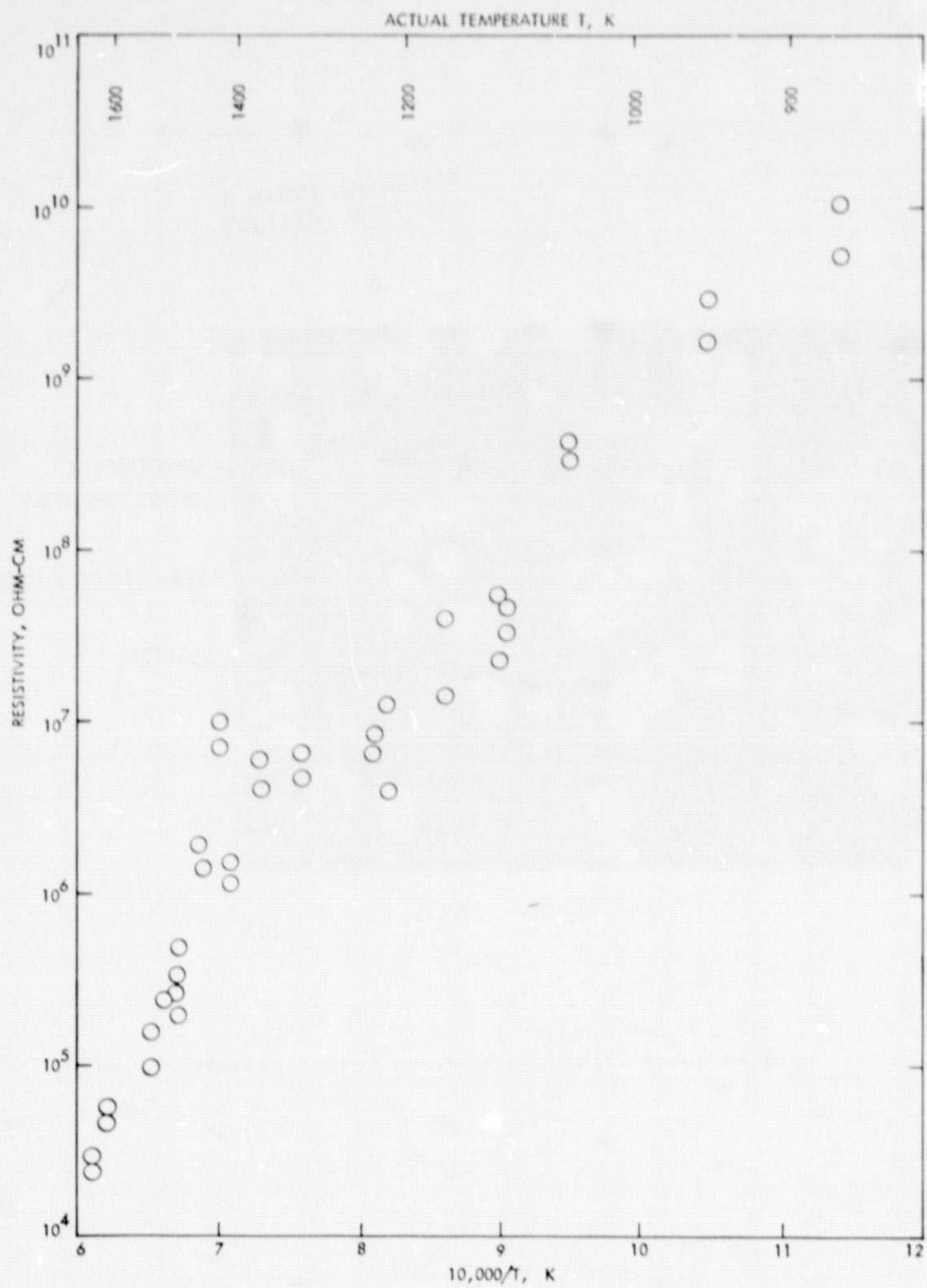


Figure 9. Electrical Resistivity of Hot Pressed 15R Polytype Sialon - DC Measurement

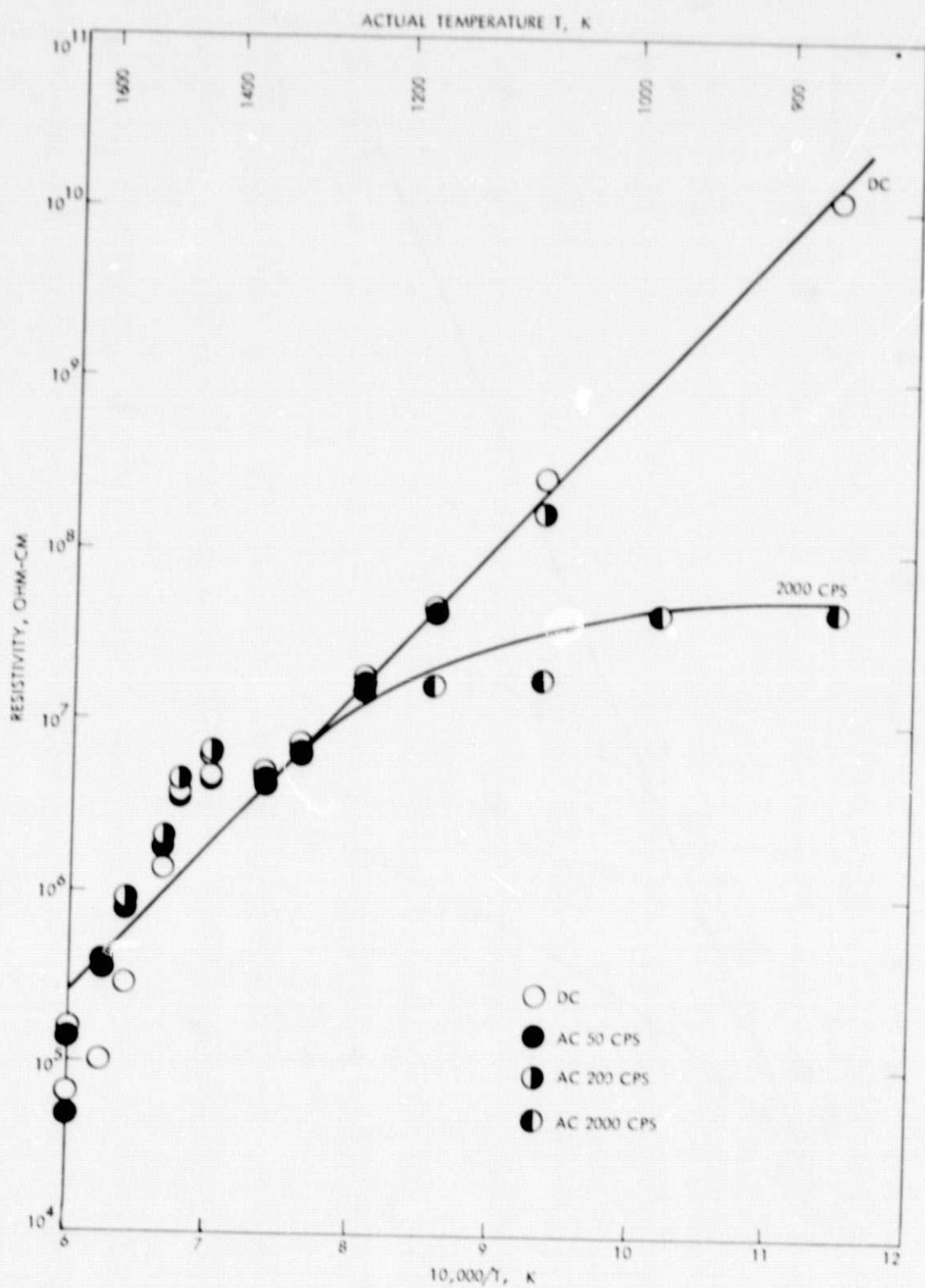


Figure 10. Electrical Resistivity of 15R Polytype Sialon Hot Pressed with Maximum Si_3N_4 Content

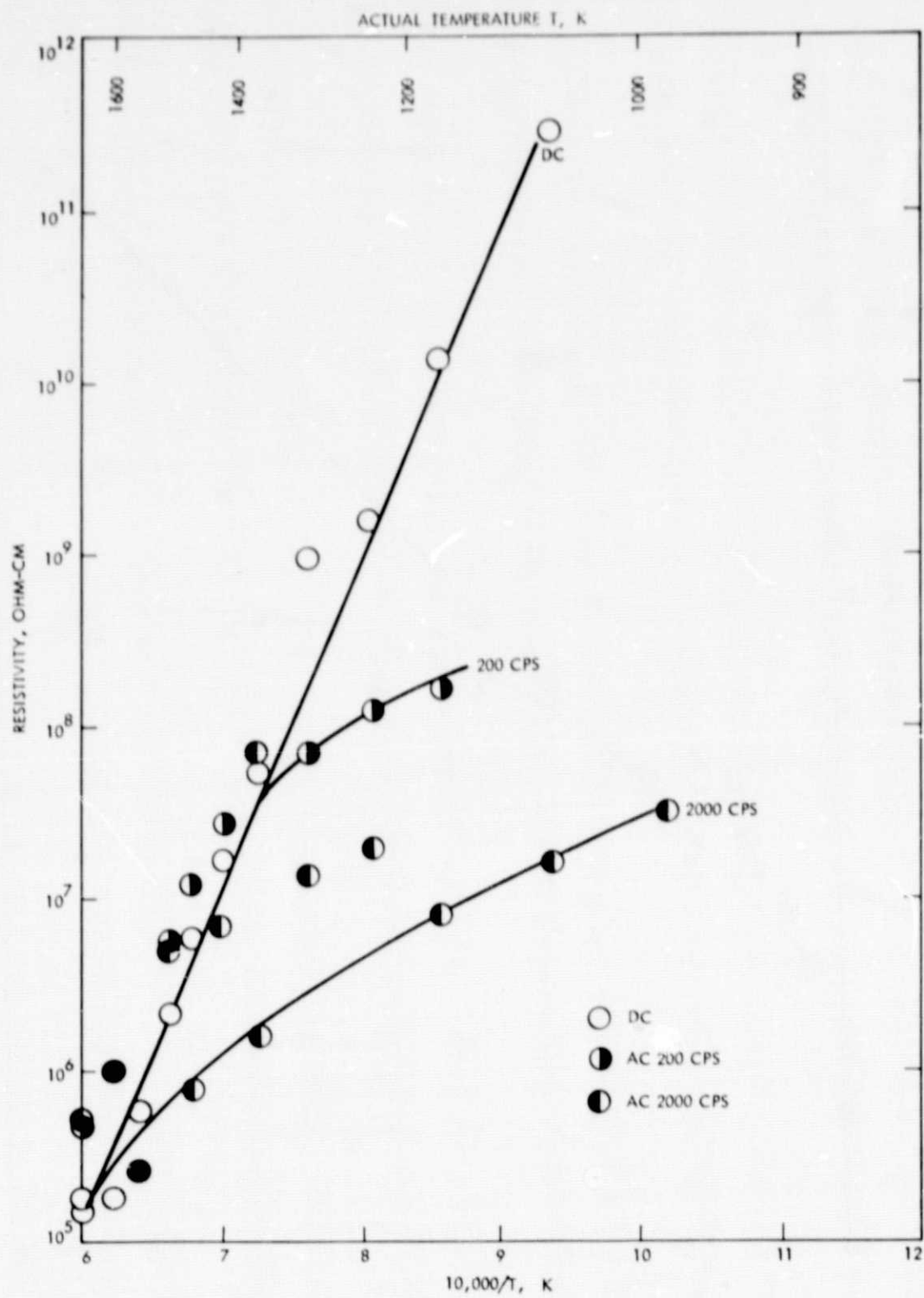


Figure 11. Electrical Resistivity of 15R Polytype Sialon Hot Pressed from SiO_2 and AlN

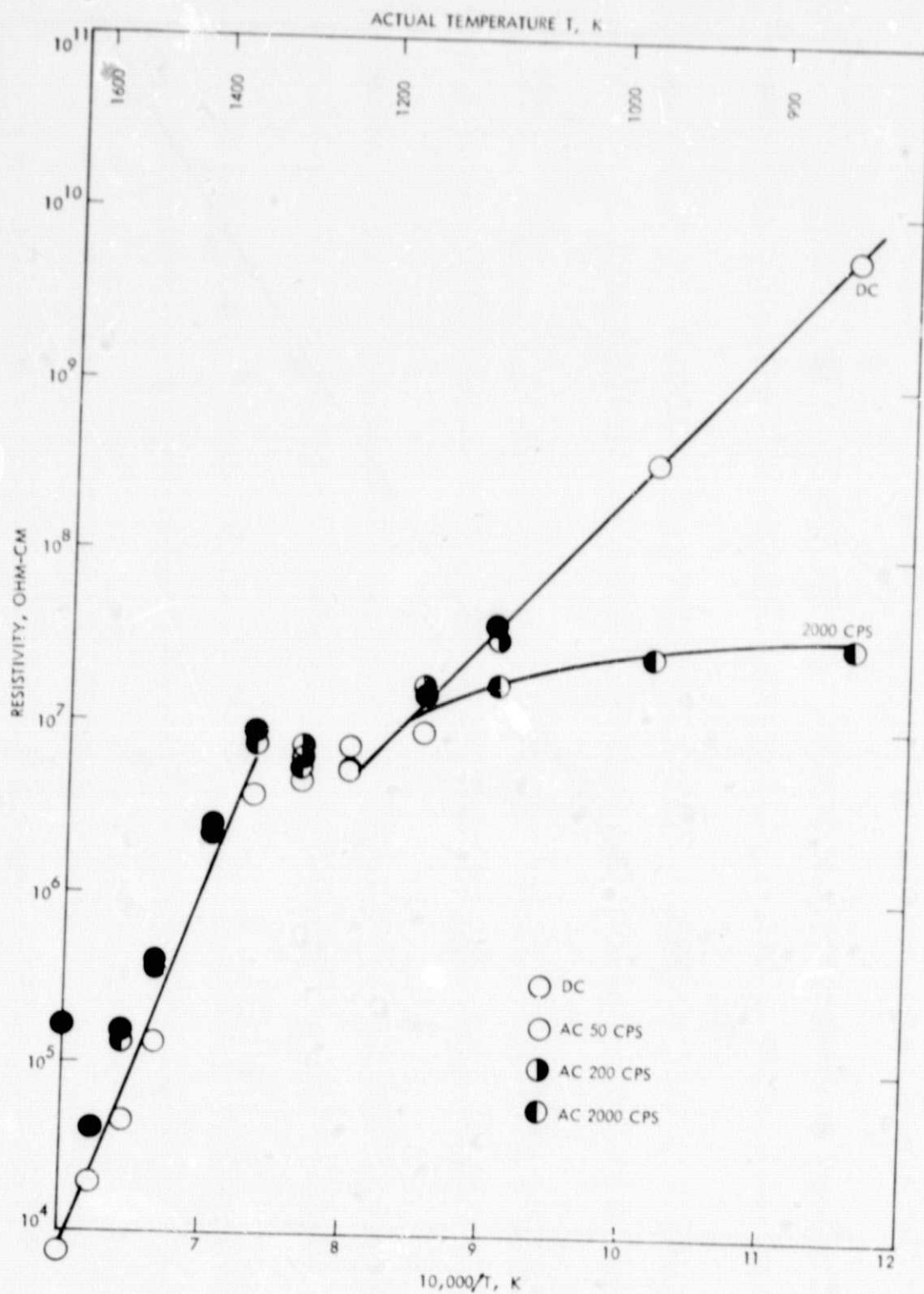


Figure 12. Electrical Resistivity of 15R Polytype SiAlon Hot Pressed with Maximum Si_3N_4 Content and 1% MgO Sintering Aid

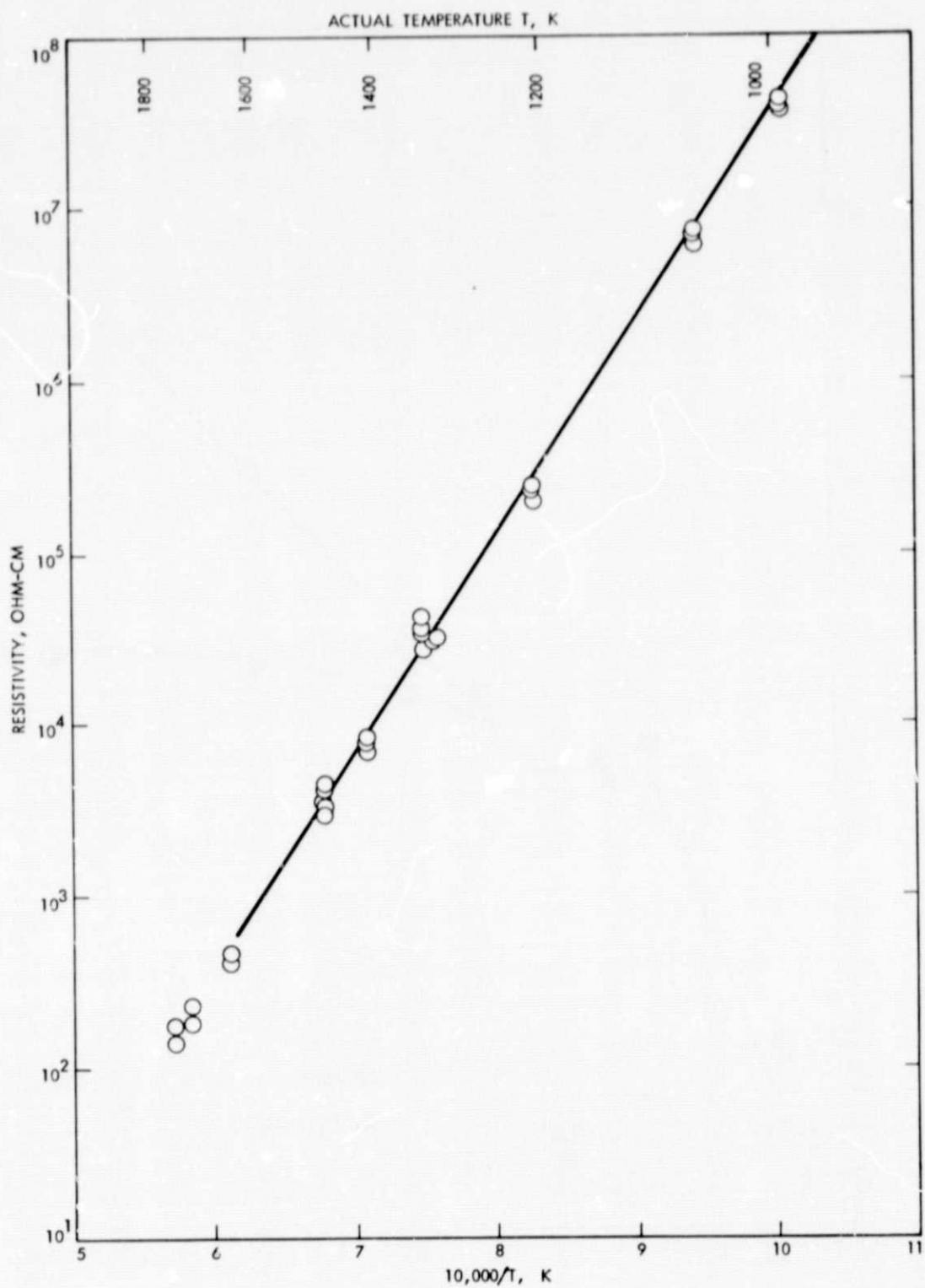


Figure 13. Electrical Resistivity of 15R Polytype Sialon Hot Pressed Between Tungsten Coated Molybdenum Plates

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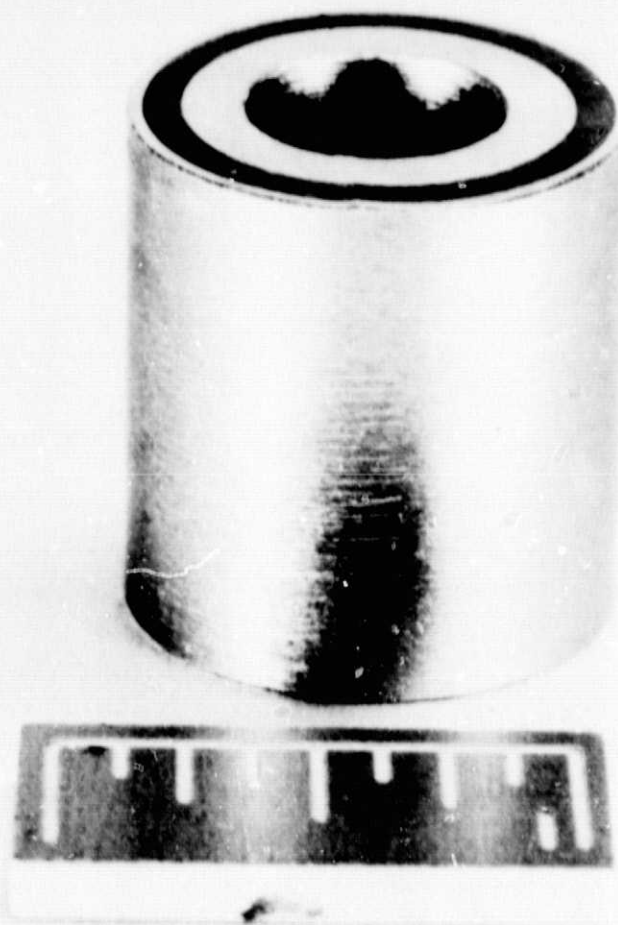


Figure 14. Section of Prototype Sialon
Insulator for NEP System